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ENVIRONMENTALLY ASSISTED CRACKING BEHAVIOR OF NICKEL ALLOYS IN SIMULATED ACIDIC AND ALKALINE GROUNDWATERS USING U-BEND SPECIMENS

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ABSTRACT

The model for the degradation of the containers for nuclear waste includes three modes of corrosion, namely general corrosion, localized corrosion and environmentally assisted cracking (EAC). The objective of the current research was to quantify the susceptibility of five nickel alloys to EAC in several environmental conditions with varying solution composition, temperature and electrochemical potential. These alloys included: Alloy 22 (N06022), Alloy C-4 (N06455), Alloy 625 (N06625), Alloy G-3 (N06985) and Alloy 825 (N08825). The susceptibility to EAC was evaluated using constant deformation (deflection) U-bend specimens in both the non-welded (wrought) and welded conditions. Results show that after more than five years exposure in the vapor and liquid phases of alkaline (pH ~ 10) and acidic (pH ~ 3) multi-ionic environments at 60°C and 90°C, none of the tested alloys suffered environmentally assisted cracking.

Keywords: high-level nuclear waste, nickel-based alloy, N06022, N06455, N06625, N06985, N08825, environmentally assisted cracking, U-bend, welded specimens, simulated acidified water (SAW), simulated concentrated water (SCW), simulated dilute water (SDW), basic saturated water (BSW)

INTRODUCTION

The current design concept for the high-level nuclear waste containers in the USA is based on a metallic multi-barrier system. This design specifies an external layer of Alloy 22 (N06022) and an internal layer or shell of type 316 stainless steel (S31603).^{1,2} The main purpose of the internal barrier is to provide structural integrity and to contribute to the shielding of radiation. The main role of the external barrier is to provide protection against corrosion. Alloy 22 was selected for the external barrier due to its excellent resistance to general corrosion, localized corrosion and environmentally assisted cracking in a

broad range of environments. ³⁻⁸ Alloy 22 is a nickel (Ni) based alloy that contains approximately 22% chromium (Cr), 13% molybdenum (Mo), 3% tungsten (W) and 3% iron (Fe). Because of its high Cr content, Alloy 22 remains passive in most industrial environments and therefore has an exceptionally low general corrosion rate. The combined presence of Cr, Mo and W imparts Alloy 22 with high resistance to localized corruptions such as pitting corrosion and crevice corrosion. After closure of the repository, the containers may suffer environmental degradation (corrosion). Dry corrosion of the waste package is expected to be negligible since the maximum temperature of the containers will be below 200°C. If water is present, there are three main aqueous corrosion mechanisms by which Alloy 22 may degrade. These include, (1) General, uniform or passive corrosion, (2) Localized corrosion (such as crevice corrosion) and (3) Environmentally assisted cracking (EAC) such as stress corrosion cracking (SCC).

The purpose of the present work was to evaluate the EAC characteristics of Alloy 22 and four other nickel alloys in several environmental conditions using constant deformation U-bend specimens. The tested solutions were concentrated versions of groundwater from the emplacement site. These solutions included: Simulated Dilute Water (SDW), Simulated Concentrated Water (SCW), Simulated Acidified Water (SAW) and Basic Saturated Water (BSW). The nickel alloys studied (in alphabetical order by UNS number) were: (1) Alloy 22 or N06022, Alloy C-4 or N06455, Alloy 625 or N06625, Alloy G-3 or N06985 and Alloy 825 or N08825.

RESISTANCE OF NICKEL ALLOYS TO ENVIRONMENTALLY ASSISTED CRACKING

From the chemical composition point of view, corrosion resistant Ni-based alloys can be grouped into five families of alloys: (1) commercially pure nickel, (2) Ni-Cu alloys, (3) Ni-Mo alloys, (4) Ni-Cr-Mo alloys and (5) Ni-Cr-Fe alloys. Results reported in this paper are for three Ni-Cr-Mo alloys (N06022, N06455 and N06625) and for two Ni-Cr-Fe alloys (N06985 and N08825). Ni-Cr-Mo alloys are the most versatile nickel alloys since they contain molybdenum for protection against corrosion under reducing conditions and chromium, which protects against corrosion under oxidizing conditions. Ni-Cr-Fe alloys in general are less resistant to corrosion than Ni-Cr-Mo alloys; however, they could be less expensive and therefore find a wide range of industrial applications such as in the production of phosphoric acid and in the handling of nitric acid. Nickel alloys are austenitic (face centered cubic), are ductile and possess a high toughness (Table 1). That is, in the absence of environmentally assisted cracking, these nickel alloys would absorb a large amount of energy before mechanical rupture.

Mill annealed Alloy 22 is highly resistant to EAC or stress corrosion cracking (SCC) in acidic concentrated chloride solutions. ⁷⁻¹³ Dunn et al. did not find SCC when they tested Alloy 22 in 14 molal Cl^- (as MgCl_2) at 110°C and 9.1 molal LiCl at 95°C under controlled potential. ⁹⁻¹² They used wedge opening loaded double cantilever beam (DCB) and compact tension (CT) specimens at stress intensities in the range 32 to 47 $\text{MPa}\cdot\text{m}^{1/2}$ for times as long as 52 weeks. ⁹⁻¹² Rebak reported that Alloy 22 U-bend specimens did not suffer SCC when exposed to 45% MgCl_2 at 154°C for up to 6 weeks. ⁷ Estil et al. performed slow strain rate tests (SSRT) at a $1.6 \times 10^{-6} \text{ s}^{-1}$ strain rate at the corrosion potential (E_{corr}) in 4 M NaCl at 98°C, saturated CaCl_2 (>10 M Cl^-) at 120°C and 1% PbCl_2 at 95°C. ¹³ None of these specimens showed a loss of ductility or secondary cracking.

Even though Alloy 22 is resistant to SCC in concentrated chloride solutions, it may be susceptible under other severe environmental conditions. ¹⁴⁻¹⁸ Andresen et al. tested the susceptibility of Alloy 22 to EAC at the corrosion potential (E_{corr}) in basic saturated water (BSW) at 110°C. ¹⁴ This BSW multi-ionic solution is a version of concentrated solutions that might be obtained after evaporative tests of

Yucca Mountain groundwaters.¹⁹ Using the reversing DC potential drop technique as a screening test, Andresen et al. reported a crack growth rate of 5×10^{-13} m/s in a 20% cold-worked specimen loaded to a stress intensity of $30 \text{ MPa}\cdot\text{m}^{1/2}$. This EAC testing was carried out in air saturated BSW water of pH ~ 13. The testing conditions used by Andresen et al. were highly aggressive and, in spite of that, the measured crack growth rate was near the detection limit of the system.¹⁴ Rebak et al. reported that Alloy 22 U-bend specimens suffered transgranular SCC when they were exposed for 336 h to aqueous solutions of 20% HF at 93°C and to its corresponding vapor phase.¹⁵ The liquid phase was more aggressive than the vapor phase.¹⁵ Pulvirenti et al. reported transgranular cracking in one out of four Alloy 22 U-bend specimen exposed for 15 days at 250°C in concentrated ground water contaminated with 0.5% lead (Pb) and acidified to pH 0.5.¹⁶⁻¹⁷ Estil et al. performed slow strain rate tests, cyclic loading tests and U-bend tests in large variety of environments (temperature, applied potential and solution composition).¹³ They only reported SCC on mill annealed (MA) Alloy 22 through SSRT in simulated concentrated water (SCW) at 73°C and at a potential of +0.3 to +0.4 V [SSC].^{13,18} When Alloy 22 was strained in SCW solution at +0.1 V [SSC], the sample did not suffer environmental assisted cracking (EAC or SCC).¹⁸ The corrosion potential (E_{corr}) of Alloy 22 in SCW solution at 60°C and 90°C was in the order of 0 to +0.1 V [SSC].²⁰ That is, it is not expected that Alloy 22 would undergo SCC in SCW solution at the free corroding potential (E_{corr}).

Recent published studies found that Alloy 22 was very resistant to stress corrosion cracking (SCC) in hot concentrated chloride solutions and in simulated concentrated water (SCW).²¹ Compact tension (CT) specimens of Alloy 22 were tested for over 3000 hours at an applied stress intensity of $47 \text{ MPa}\cdot\text{m}^{1/2}$ in 9.1 M LiCl solution at 95°C. None of the specimens suffered SCC even at applied potentials higher than the crevice repassivation potential.²¹ A similar test was run on an Alloy 22 CT specimen at an applied potential of 380 mV [SCE] in SCW solution at 73°C and 95°C. The specimen was free from SCC.²¹ The same investigators reported that Alloy 22 U-bend specimens did not crack in presence of supersaturated PbCl₂ pH 0.5 at 95°C after more than 40 days of testing.²¹

Similar to the results reported above in detail for N06022, the other four studied nickel alloys (N06455, N06625, N06985 and N08825) are also highly resistant to environmentally assisted cracking (EAC). For example, it was reported that Alloy 825 did not suffer EAC when it was tested by slow strain rate testing in 9.1 molal LiCl solution at 110°C or in 5.8 molal NaCl solution containing 0.01 M sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) at 95°C both at the corrosion potential (E_{corr}) and under anodic polarization.⁶ EAC of Alloy 825 was only observed in 14 molal chloride (40% MgCl₂) solution at 120°C.⁶

Environmentally assisted cracking (EAC) data of the other nickel alloys studied here are scarce. Nickel alloys may suffer EAC during oil and gas production from sour wells containing high chloride at temperatures above 200°C. Generally for these applications, nickel alloys are heavily cold worked to purposely increase their strength. NACE standard MR0175 specifies the hardness limit for the nickel alloys used in oil and gas production. For example, the maximum allowed hardness for N06625 is 35 HRC, for N06985 is 39 HRC and for N08825 is 35 HRC. In the mill-annealed (MA) condition, the hardness of Ni-Cr-Mo and Ni-Cr-Fe alloys is approximately 90 HRB (Table 1). Depending on their corrosion resistance in sour well applications, the above-mentioned nickel alloys have been ranked as follows: N06625 and N06985 higher than N08825.²² At temperatures above 150°C, nickel alloys may also suffer EAC in caustic aqueous environments such as 50% NaOH solutions.^{8,22} Another environment that may promote EAC in nickel alloys is hot wet hydrofluoric acid.^{7-8,15,22}

EXPERIMENTAL

Laboratory testing for environmentally assisted cracking is commonly carried out using a variety of specimens and techniques. The techniques are usually grouped by the way the mechanical stress is applied to the test specimen. The most common tests include: (a) Constant deformation, (b) Constant Load and (c) Slow strain rate tests. In order to better simulate the likely field behavior, the specimens (technique) that are used for laboratory testing should closely reproduce the field conditions. A metallic static container resting horizontally may contain only mechanical residual stresses due to fabrication (e.g. welding) or possibly rock fall impact, which would produce stored strain energy or cold work. That is, the most representative specimens for laboratory testing would be the constant deformation types such as U-bend, which contain residual stresses due to permanent bending.

The studied nickel alloys included Alloy 22 (N06022), C-4 (N06455), Alloy 625 (N06625), G-3 (N06985) and Alloy 825 (N08825). Table 2 shows the compositions of the studied alloys. The welded U-bend specimens had matching filler metal, that is, a wire of the same alloy was used to produce the welds, except in the case of alloys 825 and C-4 in which Alloy 22 wire was used for the weld (Table 2). In the designation of the specimens, the first letter corresponded to the type of alloy. Thus an initial letter D represents Alloy 22, the letter C represents C-4, the letter L represents Alloy 625, the letter B represents G-3 and the letter A represents Alloy 825. The second letter in the designation represents the type of specimen, in this case the letter U represents U-bend. The third letter designates if the material for the U-bend was seamless wrought mill annealed (MA) (letter A) or had a weld seam (letter different from A) (Figure 1). These three letters are followed by a three-digit serial number. Thus, BUC049 is the U-bend specimen number 49, with a weld seam and made of Alloy G-3.

The U-bend specimens were machined from sheet stock. The specimens were tested in the as-machined condition, which corresponded to surface finish with a root mean square (RMS) roughness of 32μ -inch. This surface roughness roughly corresponds to a 120-grit paper, a standard industrial finish for corrosion testing. The U-bend specimens were prepared using $3/4$ -inch (~ 19 mm) wide and $1/16$ -inch (~ 1.6 mm) thick strips according to ASTM G 30.²³ The resulting specimen had a constant nominal separation between both legs, or ends, of 0.5 inch (~ 13 mm) secured by a bolt, which was electrically insulated from the specimen through ceramic zirconia washers (Figure 2). The total plastic deformation in the external outer fiber was approximately 12%. Single U-bends were produced using both wrought sheets and welded sheets. In the welded specimens, the weld was across the apex of the bend (Figure 1). The weld process was gas metal arc welding (GMAW) using filler metal and the seam had full penetration. Typical mechanical properties of MA sheet material are listed in Table 1. Table 2 lists the chemical composition of the sheet material and the filler metal used for the fabrication of the U-bend specimens. These specimens were degreased in acetone before testing.

The immersion testing was carried out in the Long Term Corrosion Test Facility (LTCTF) at Lawrence Livermore National Laboratory (LLNL). The testing was carried out following general procedures for laboratory immersion testing given in ASTM G 31.²³ The testing electrolyte solutions for the U-bend were solutions containing several ionic species. The volume of the electrolyte in each vessel was approximately 1000 liters. Table 3 shows the composition of the multi-component electrolyte solutions mentioned in this paper. Table 3 also shows the composition of the water from well J-13 near Yucca Mountain. The solutions used in this study are concentrated versions of J-13 water. For example, SDW (Simulated Dilute Water) is approximately 10 times more concentrated than J-13 water and has a pH ~ 10 , SCW (Simulated Concentrated Water) is approximately 1000 times more concentrated than J-13 water (pH ~ 10) and SAW (Simulated Acidified Water) is also approximately 1000 times more concentrated.

trated than J-13 water but acidified to pH ~3. The U-bend immersion tests were carried out at 60°C and 90°C. Roughly half of the tested specimens were exposed to the liquid phase of the solution and the other half to the vapor phase, where condensation occurred over the specimens. The reported temperature corresponded to the liquid phase. The exposure time was slightly over 5 years (the actual exposure time is given in Table 4). Two Alloy 22 specimens were tested in the liquid phase of Basic Saturated Water (BSW) at 105°C. The testing time was 1149 days for the double U-bends specimen (ARC22U20A + ARC22U20B) and 742 days for the single U-bends specimen (DUB163). The testing electrolyte solutions were naturally aerated; that is, the solutions were not purged with any gas; however the ingress of air above the solution level was not restricted. All tests were carried out under ambient pressure. After testing, the samples were evaluated using standard procedures such as optical and scanning electron microscopy.

EXPERIMENTAL RESULTS AND DISCUSSION

Single U-bend Specimens from LTCTF

The single U-bend specimens were exposed to three different multivalent ionic electrolyte solutions in the Long Term Corrosion Test Facility (LTCTF) at the free corrosion potential (E_{corr}) for more than 5 years. Two of these electrolyte solutions (SCW and SDW) were alkaline of pH ~10 and one electrolyte (SAW) was acidic of pH ~3 (Table 3). Two hundred seventy six (276) specimens were removed from six of the testing tanks, rinsed in deionized water and allowed to dry in the laboratory atmosphere. Data regarding performance of 52 Alloy 22 specimens were reported before.²⁴ Table 4 lists the specimens by their label, by the vessel they were exposed to and by the length of time they were tested. The specimens were labeled starting with a characteristic letter to identify the alloy. These letters were: D for Alloy 22, C for C-4, L for 625, B for G-3 and A for Alloy 825. The second letter was U for U-bend specimen. The third letter identifies if the specimen is seamless or contains a weld. If the third letter is A, the specimen is seamless, if the third letter is different from A, the specimen contains a weld seam in the apex of the specimen (Figure 1). Table 2 shows that matching filler metal was used to produce the welds of specimens for Alloy 22, 625 and G-3. For the specimens made of C-4 and Alloy 825, Alloy 22 material was used for the filler metal. The heat numbers and composition for both the base material sheet and the filler metal are given in Table 2. In general, three specimens were examined for each temperature, solution composition and metallurgical condition.

The 276 specimens (Table 4) were individually examined optically in a stereomicroscope using up to 100 times magnification. Figure 2 shows the macroscopic appearance of two of the examined specimens, one welded and one non-welded. The principal characteristics of this individual examination are given in Table 5. A few selected specimens were also studied in a scanning electron microscope and others were mounted for metallographic sectioning. Stereomicroscope studies showed that most of the specimens were completely featureless, that is, they appeared shiny metallic similar to the non-tested condition (Table 5). Most of the specimens had deposits of crystals (salts) from the electrolyte. The specimens that were exposed to the vapor phase had a lower amount of deposits than the specimens exposed to the liquid phase. However, surface features suggest that the specimens exposed to the vapor phase had abundant condensation on them. The specimens that were tested at the higher temperature (90°C) in the liquid phase in general showed a higher degree of discoloration than the specimens tested at 60°C. This may suggest that there was more interaction between the specimens and the environment at the higher temperature; however, most of the colors and deposits observed (Table 5) suggest that these were the result of buildup from the environment rather than due to a reaction of the metal with the environment.

ronment. The origin of the colors (e.g. golden/green/blue) is not yet known. The golden color was probably caused by the deposit of little crystals of this color on the surface. Some of these small crystals deposited from the SAW solution are rich in iron. Studies of the scales and oxide films on the Alloy 22 specimens are reported separately.²⁵ An important observation from Table 5 is that none of the 276 examined specimens showed any indication of corrosion or environmental cracking (EAC).

Scanning electron microscopy (SEM) studies showed that some of the specimens had microcracks on their convex surface, perpendicularly to the applied mechanical stress. These cracks were shallow (approximately 2 μ m deep) and less than 0.1 mm in length. Since these microcracks were also observed in non-tested specimens, it can be concluded that the microcracks in the tested specimens existed before these specimens were immersed in the testing electrolytes in 1997. Figure 3 shows two SEM images of the examined specimens (Table 4). None of them suffered cracking (EAC).

Single U-bend and Double U-bend Specimens from Bench Top Testing

Two U-bend Alloy 22 (N06022) specimens removed from BSW solution (pH ~ 13) at 105°C after 1149 days (27,576 h) and 742 days (17,808 h) of testing were also free from corrosion or environmentally assisted cracking.

ENVIRONMENTALLY ASSISTED CRACKING AND THE CORROSION POTENTIAL

Table 6 shows the corrosion potential (E_{corr}) of Alloy 22 and platinum in the electrolyte solutions mentioned in this report. These are E_{corr} values in normally aerated solutions. Table 6 is an updated version of a table published before.²⁰ The E_{corr} values shown for Alloy 22 could also be considered relevant for the other nickel alloys (C-4, 625, G-3 and 825) since all these alloys would contain a chromium oxide film on their surfaces (the amount and protectiveness of chromium oxide in the film would depend on the pH of the solution). First of all, Table 6 shows that the E_{corr} of Alloy 22 is highly stable in all the tested solutions. That is, E_{corr} has not considerably changed over the last year of testing. The most typical values of E_{corr} for Alloy 22 in SCW and SDW at 60°C and 90°C and for BSW at 105°C were in the vicinity of 0 V to +0.1 V [SSC] (Table 6). For SAW, E_{corr} for Alloy 22 was higher, in the order of +0.3 to +0.4 V [SSC] (Table 6). The higher E_{corr} in the acidic solution could be a consequence of the formation of a more stable chromium oxide film on the surface. In the higher pH solutions, nickel oxides should be more stable than chromium oxide.²⁶ That is, specimens of five nickel alloys with residual stresses due to constant deformation, were tested in multi-ionic solutions in a wider range of pH (3 to 13) and potential (0 to +0.4 V) and none of them suffered environmentally assisted cracking (EAC).

RELATIVE CORROSION RESISTANCE OF THE STUDIED NICKEL ALLOYS

The relative resistance to environmentally assisted cracking (EAC) of five nickel alloys in multi-ionic solutions simulating concentrated ground water is reported here. None of these alloys suffered EAC in the tested environments. However, it is relevant to note that there are considerable differences in the corrosion behavior of these five nickel alloys when they are tested in other (more aggressive) conditions. Table 7 and Figures 4-7 show the corrosion rates of the five nickel alloys of interest in a variety of conditions. Figure 4 shows that in the highly aggressive green death solution, the lowest corrosion rate corresponded to Alloy 22. The corrosion rate of the other nickel alloys (C-4, 625, G-3 and 825) was more than two orders of magnitude higher than for Alloy 22. Green death solution contains approx

mately 0.6 M Chloride, it is a highly acidic and highly oxidizing and therefore it promotes localized corrosion in many commonly passive alloys. Figure 4 shows that Alloy 22 was the only one of the five tested nickel alloys that did not suffer localized corrosion when tested in boiling green death solution. Alloy 22 contains the appropriate amount of beneficial elements for protection against localized corrosion (e.g. high chromium, high molybdenum and tungsten) (Table 2). Figure 5 shows the corrosion rate of the same five alloys in boiling 10% nitric acid, which is oxidizing. Since chloride ions are not present, alloys that contain a large amount of chromium (and even iron) would perform well under oxidizing conditions. Figure 5 shows that all alloys containing over 20% chromium had the lowest corrosion rates. The highest corrosion rate in nitric acid corresponded to Alloy C-4, which contains 16% chromium (Table 2). Figures 6 and 7 show the corrosion rate of the five nickel alloys under acidic reducing conditions. Figure 6 shows that in boiling 10% sulfuric acid, alloys containing a small amount of copper (besides molybdenum) perform well in hot sulfuric acid (Table 2). That is, even though alloy 825 and G-3 had lower content in molybdenum than C-4, they had similar corrosion rates. Figure 7 shows that the lowest corrosion rate in boiling hydrochloric acid corresponded to C-4, the alloy that contains the higher amount of the beneficial element molybdenum (Table 2). Overall, Table 7 and Figures 4-7 show that in most environments (reducing and oxidizing) Alloy 22 has one of the lowest corrosion rates compared to the other four alloys. The data support the argument for the superior corrosion resistance of Alloy 22 as compared to the other industrially widely used nickel alloys.

FINAL REMARKS

None of the tested nickel alloys suffered environmentally assisted cracking (EAC) under the tested conditions reported in this paper. Therefore, the environment was not too aggressive or the alloys were too resistant. It is known that the tested environments are aggressive enough to cause cracking in welded U-bend of TiGr 12 (R53400) alloy at the corrosion potential.²⁷ Also, the tested nickel alloys are not equally resistant to corrosion. General and localized corrosion rate data above show that they behave differently from each other under more aggressive conditions. Since four alloys, which are less corrosion resistant than Alloy 22, did not fail by EAC after 5 years immersion in hot solution equivalent to concentrated ground water, the corollary is that Alloy 22 has an even a larger margin of safety in the tested environments.

CONCLUSIONS

- (1) Mill annealed (MA) and welded nickel alloys (N06022, N06455, N06625, N06985 and N08825) are highly resistant to environmentally assisted cracking (EAC) in multi-ionic solutions that may represent concentrated versions of ground water at Yucca Mountain.
- (2) U-bend specimens exposed at E_{corr} for 5 years in SAW, SCW and SDW solutions at 60°C and 90°C and in BSW solution at 105°C for 3 years were free from EAC.
- (3) The nickel alloys were resistant to EAC at pH ~ 3 with E_{corr} of approximately +0.3 to 0.4 V [SSC] (SAW) and pH ~ 10 to 13 with E_{corr} of approximately 0 to +0.1 V [SSC] (SCW, SDW and BSW).
- (4) Even though none of the tested nickel alloys suffered EAC, it is known that the overall corrosion resistance of Alloy 22 is much higher than for the other four alloys.

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TABLE 1
TYPICAL MECHANICAL PROPERTIES OF NICKEL ALLOYS SHEET
AT AMBIENT TEMPERATURE

Alloy, UNS	First Letter Notation for Specimens	Tensile Strength [UTS] (MPa)	Yield Stress [0.2%] (MPa)	Elongation to Rupture (%)	Hardness (RB)
Alloy 22, N06022	D	800	407	57	93
C-4, N06455	C	768	416	52	90
Alloy 625, N06625	L	910	468	47	94
G-3, N06985	B	724	348	48	--
Alloy 825, N08825	A	758	421	39	--

TABLE2
CHEMICALCOMPOSITIONANDHEATNUMBERSOFTHEU -BENDSPECIMENS(Wt%)
THEALLOYSAREORGANIZEDALPHABETICALLYBYUNS NUMBER

	UNS	Heat	Ni	Cr	Mo	W	Fe	Others
Alloy22, Base	N06022	2277-0-3264	57	21.3	13.4	2.9	4.4	1.14Co,0.29Mn,0.17 V
WeldFiller	N06022	2277-4-3263	57	21.6	13.5	2.9	3.6	0.89Co,0.32Mn,0.15 V
C-4,Base	N06455	6455-5-0906	68	15.43	15.66	---	0.29	0.21Mn,0.21Ti
WeldFiller	N06022	2277-4-3263	57	21.6	13.5	2.9	3.6	0.89Co,0.32Mn,0.15 V
Alloy625, Base	N06625	VX1178AK	61.13	21.88	9.16	---	3.72	3.52Cb+Ta,0.29Al, 0.17Ti
WeldFiller	N06625	53738	65.1	21	8.47	---	0.75	3.4Cb+T a,0.26Al, 0.29Ti
G-3,Base	N06985	Z3896HG	46.41	21.72	6.73	0.89	19.4	0.77Mn,0.21Si,1.77 Cu,1.86Co,0.2 Cb+Ta
WeldFiller	N06985	Z0708HG	46.29	21.14	6.68	0.84	19.94	0.76Mn,0.34Si,1.85 Cu,2.05Co,
Alloy825, Base	N08825	HH7588FG	44.06	22.98	3.05	---	26.67	0.39Mn,0.17Si,1.71 Cu,0.88Ti
WeldFiller	N06022	2277-4-3263	57	21.6	13.5	2.9	3.6	0.89Co,0.32Mn,0.15 V

TABLE3
CHEMICALCOMPOSITIONOFTHEELECTROLYTESOLUTIONS(mg/L)

Ion	SDW pH10.1	SCW pH10.3	SAW pH2.8	BSW pH13	J-13WellW ater pH7.4
K ⁺	34	3400	3400	81,480	5.04
Na ⁺	409	40,900	40,900	231,224	45.8
Mg ²⁺	1	<1	1000	---	2.01
Ca ²⁺	0.5	<1	1000	---	13
F ⁻	14	1400	0	1616	2.18
Cl ⁻	67	6700	24,250	169,204	7.14
NO ₃ ⁻	64	6400	23,000	177,168	8.78
SO ₄ ²⁻	167	16,700	38,600	16,907	18.4
HCO ₃ ⁻	947	70,000	0	107,171	128.9
SiO ₂ (aq)	~40	~40	~40	9038	61.1

TABLE4
CONSTANT DEFORMATION (U -BEND) TESTS OF NICKEL ALLOYS
LIST OF EXAMINED SPECIMENS

	SAW, 60°C	SAW, 90°C	SCW, 60°C	SCW, 90°C	SDW, 60°C	SDW, 90°C
Vessel	25	26	27	28	29	30
Date in	06Feb1997	21Feb1997	10Mar1997	10Apr1997	14Apr1997	05Jun1997
Date out	20May2002	21May2002	17May2002	22May2002	10May2002	22May2002
Exp. Time, days(h)	1930 (46,320h)	1916 (45,984h)	1895 (45,480 h)	1869 (44,856h)	1853 (44,472h)	1813 (43,512h)
Specimens Wrought and Welded	DUA019-021 DUB019-021 CUA019-021 CUB019-021 LUA019-021	DUA049-051 DUB049-051 CUA049-051 CUB049-051 LUA049-051	DUA079-081 DUB079-081 CUA079-081 CUB079-081 LUA079-081	DUA109-111 DUB109-111 CUA109-111 CUB109-111 LUA109-111	DUA127 DUB127 CUA127 CUB127 LUA127	DUA139 DUB139 CUA139 CUB139 LUA139
Vapor Phase	LUJ019-021 BUA019-021 BUC019-021 AUA019-021 AUB019-021	LUJ049-051 BUA049-051 BUC049-051 AUA049-051 AUB049-051	LUJ079-081 BUA079-081 BUC079-081 AUA079-081 AUB079-081	LUJ109-111 BUA109-111 BUC109-111 AUA109-111 AUB109-111	LUJ127 BUA127 BUC127 AUA127 AUB127	LUJ139 BUA139 BUC139 AUA139 AUB139
Specimens Wrought and Welded	DUA022-024 DUB022-024 CUA022-024 CUB022-024 LUA022-024	DUA052,054 DUB053,054 CUA052-054 CUB052-054 LUA052-054	DUA082-084 DUB082-084 CUA082-084 CUB082-084 LUA082-084	DUA112,114 DUB113,114 CUA112-114 CUB112-114 LUA112-114	DUA128 DUB128 CUA128 CUB128 LUA128	DUA140 DUB140 CUA140 CUB140 LUA140
Liquid Phase	LUJ022-024 BUA022-024 BUC022-024 AUA022-024 AUB022-024	LUJ052-054 BUA052-054 BUC052-054 AUA052-054 AUB052-054	LUJ082-084 BUA082-084 BUC082-084 AUA082-084 AUB082-084	LUJ112-114 BUA112-114 BUC112-114 AUA112-114 AUB112-114	LUJ128 BUA128 BUC128 AUA128 AUB128	LUJ140 BUA140 BUC140 AUA140 AUB140
Total Examined Specimens	60	58	60	58	20	20
Specimens that suffered EAC	0	0	0	0	0	0

TABLE5

STEREOMICROSCOPE OBSERVATIONS OF THE TETRAFLUOROETHYLENE (TFE) -BEND SPECIMENS

Conditions	VaporPhase	LiquidPhase
Vessel25 SAW,60°C	Shiny metallic. Few isolated brown deposits. No corrosion or cracking	Shiny gray-green-blue. Brown deposits mostly in concave area. No corrosion or cracking
Vessel26 SAW,90°C	Shiny metallic or light gray. Brown deposits in concave area. No corrosion or cracking	Dark golden with green patches. Abundant brown deposits in concave area. No corrosion or cracking
Vessel27 SCW,60°C	Shiny metallic and dull light gray with bluish and golden patches. Some white deposits. No corrosion or cracking	Shiny metallic or light golden. Some white deposits in concave area. No corrosion or cracking
Vessel28 SCW,90°C	Shiny dark gray and golden. Little white and green deposits in concave area. No corrosion or cracking	Sample covered by white salt-like deposits. Underneath deposits shiny light golden. No corrosion or cracking
Vessel29 SDW,60°C	Shiny metallic light gray. Very little deposits. No corrosion or cracking	Shiny metallic light gray. Little white deposits. No corrosion or cracking
Vessel30 SDW,90°C	Shiny metallic. No deposits. No corrosion or cracking	Shiny metallic. White deposits in concave area. No corrosion or cracking

TABLE6
CORROSIONPOTENTIALOFALLOY22INMULTIIONICSOLUTIONS

Cell#	SampleT ypeandNu mber	InitialConditionoftheSa m- ple	E _{corr} Day 1[SSC]	E _{corr} 31Aug02 [SSC]	E _{corr} 31Aug03 [SSC]
CELL1 :Environment:SAWfromLTCTF,60°C.StartingDate:13April2001					
1	Alloy22U -bendDUB028	1527days(4+yr)inLTCTF	0.178	0.385	0.387
1	Alloy22U -bendDUB157	Untested,600grit	0.432	0.403	0.412
1	PtlatinumrodWEA007	Untested,600grit	0.461	0.415	0.427
CELL2:Enviro nment:SAWfromLTCTF,90°C.StartingDate:13April2001					
2	Alloy22U -bendDUB052	1512days(4+ yr)inLTCTF	0.386	0.276	0.289
2	Alloy22U -bendDUB159	Untested,600grit	0.362	0.299	0.382
2	PlatinumrodWEA006	Untested,600grit	0.419	0.383	0.382
CELL7:Enviro nment:SCWfromLTCTF,60°C.StartingDate:13April2001 ^(A)					
7	Alloy2 2U -bendDUB088	1495days(4+yr)inLTCTF	0.070	0.019	---
7	Alloy22U -bendDUB156	Untested,600grit	-0.039	-0.014	---
7	PlatinumrodWEA010	Untested,600grit	-0.032	0.032	---
CELL3:Enviro nment:SCWfromLTCTF,90°C.StartingDate:13April 2001					
3	Alloy22U -bendDUB112	1464days(4+yr)inLTCTF	-0.027	0.000	-0.026
3	Alloy22U -bendDUB161	Untested,600grit	-0.161	-0.061	-0.063
3	PlatinumrodWEA003	Untested,600grit	-0.050	0.069	0.062
CELL5:Enviro nment:SDWfromLTCTF, 60°C.StartingDate:13April2001					
5	Alloy22U -bendDUB128	1460days(4+yr)inLTCTF	0.077	0.025	0.051
5	Alloy22U -bendDUB150	Untested,600grit	-0.082	-0.067	0.016
5	PlatinumrodWEA011	Untested,600grit	0.179	0.258	0.236
CELL6: Environment:SDWfromLTCTF,90°C.StartingDate:13April2001					
6	Alloy22U -bendDUB132	1457days(4+yr)inLTCTF	0.032	0.081	0.083
6	Alloy22U -bendDUB162	Untested,600grit	-0.096	0.082	0.103
6	PlatinumrodWEA005	Untested,600grit	0.138	0.074	0.137
CELL4:Enviro nment:BSWfromBenchTop,105°C.StartingDate:26April2001 ^(B)					
4	Alloy22Do ubleU -bend ARC22U20A+ARC22U20B	407days(1+yr)BenchTop	-0.112	0.046	0.048
4	Alloy22U -bendDUB163	Untested,600grit	-0.754	0.027	-0.002
4	PlatinumrodWEA014	Untested,600grit	0.030	0.074	0.087

(A) Teststoppedon18Nov01

(B) Teststoppedon25Apr03

TABLE 7
TYPICAL CORROSION RATES IN MPY OF SELECTED NICKEL ALLOYS
IN BOILING SOLUTIONS
DATA FROM REFERENCE 8 AND FROM HAYNES INTERNATIONAL

Alloy, UNS	Green Death (11.5% H_2SO_4 + 1.2% HCl + 1% FeCl_3 + 1% CuCl_2)	10% Nitric Acid (HNO_3)	10% Sulfuric Acid (H_2SO_4)	2.5% Hydrochloric Acid (HCl)
Alloy 22, N06022	2.8	0.42	9.56	137.9
C-4, N06455	890	6.39	20.24	77.43
Alloy 625, N06625	1650	0.36	36.6	619
G-3, N06985	1653	0.38	17.95	341
Alloy 825, N08825	1977	0.47	20.2	360

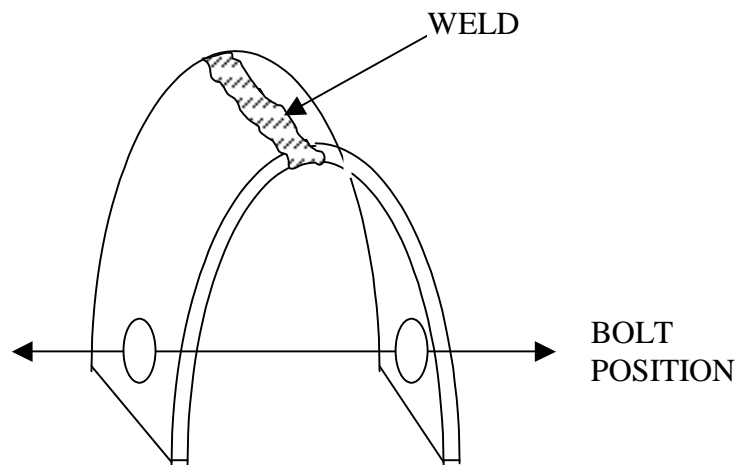


FIGURE 1: Schematic representations showing the position of the weld in the U-bend specimens.

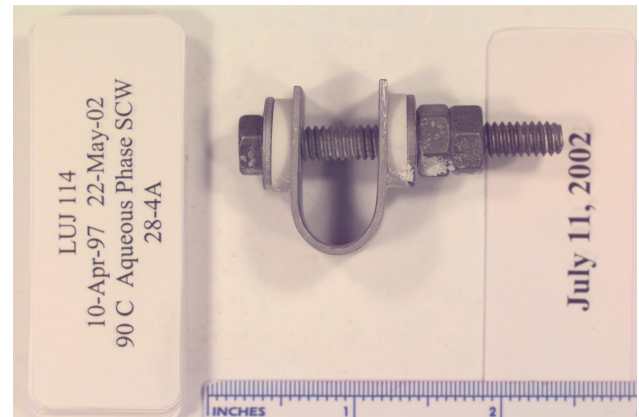
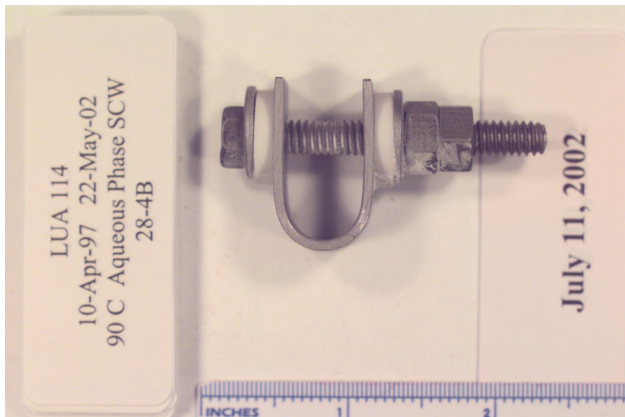


FIGURE2:Macrographoftwoofthe276examinedU-bendspecimens.BothareforAlloy625,leftthe non-weldedLUA114andrighttheweldedLUJ114exposedtoliquidSCWat90°Cfor5years(Table4). Noneofthespecimenssufferedcracking(EAC).

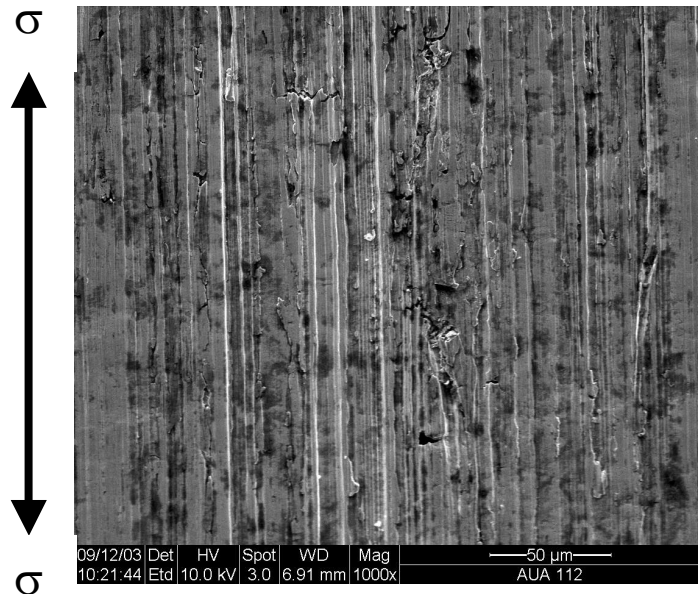
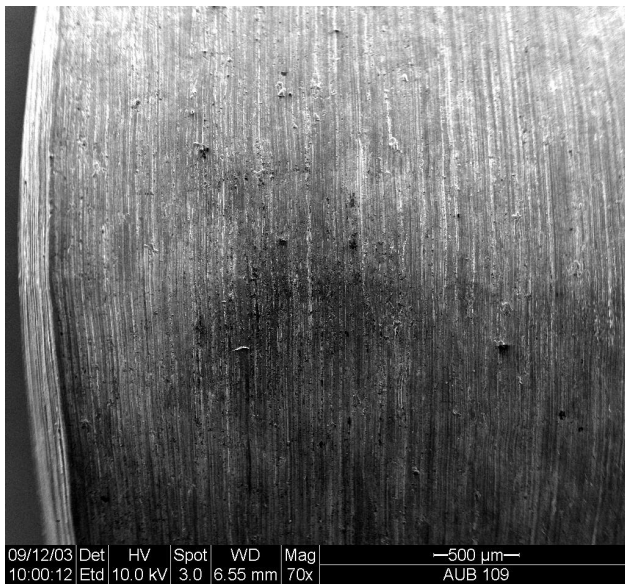


FIGURE3:SEMImagesoftwoofthe276examinedU-bendspecimens.BothareforAlloy825,leftthe weldedAUB109(X70magnification)andrightthenon-weldedAUA112(X1000magnification)both exposedtoliquidSCWat90°Cfor5years(Table4).Thearrowshowsthe directionoftheapplied stresses.Noneofthespecimenssufferedcracking(EAC).

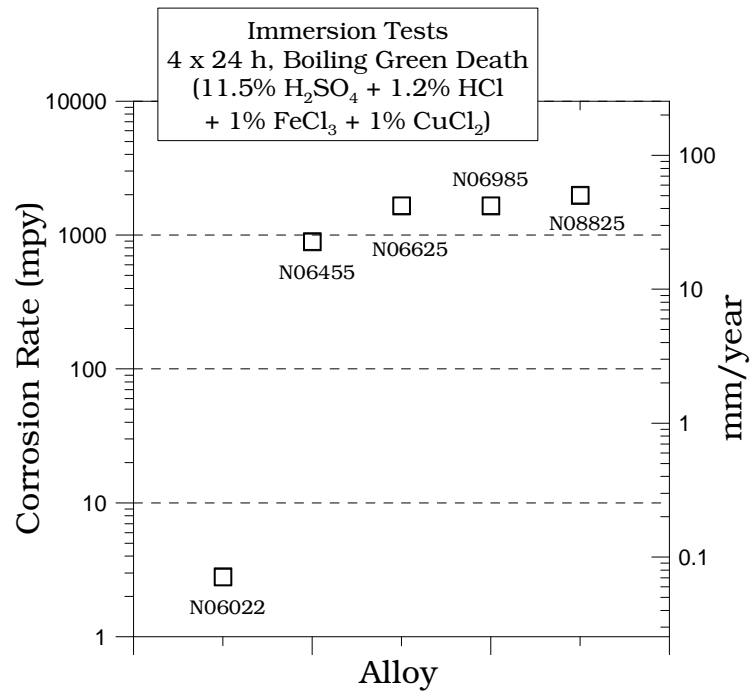


FIGURE4:CorrosionRateofthefivenickelalloysinboilinggreendeathsolution.

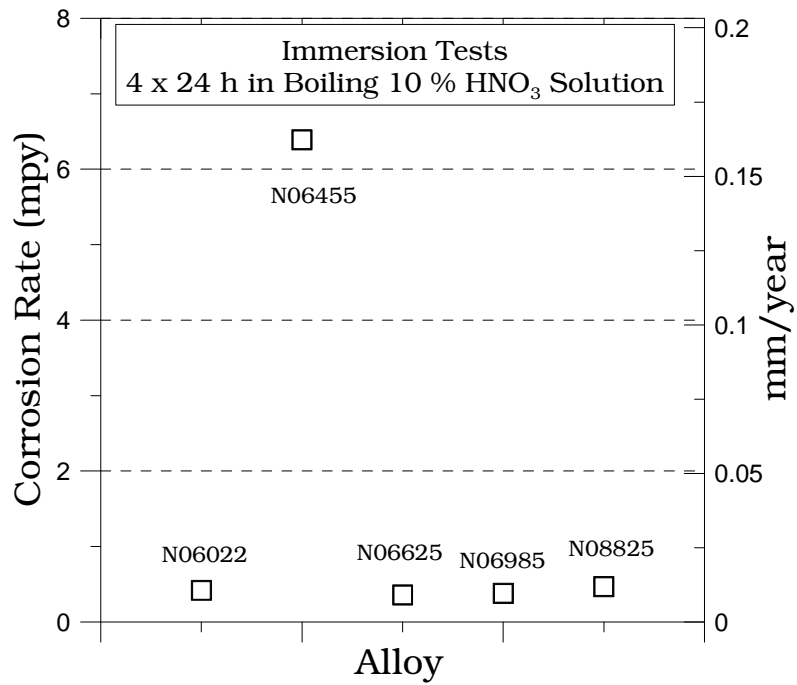


FIGURE5:CorrosionRateofthefivenickelalloysinboiling10%nitricacidsolution.

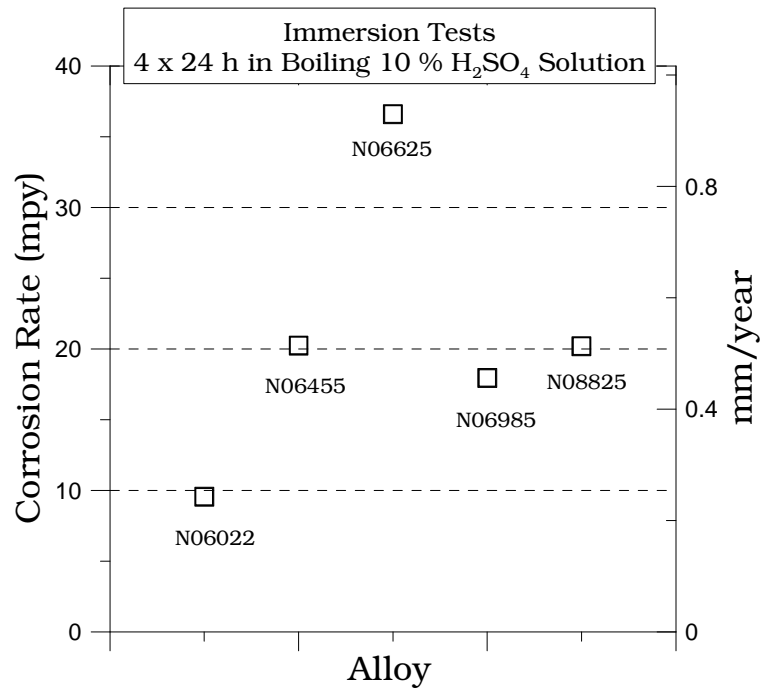


FIGURE6:CorrosionRateofthefivenickelalloysinboiling10%sulfuricacidsolution.

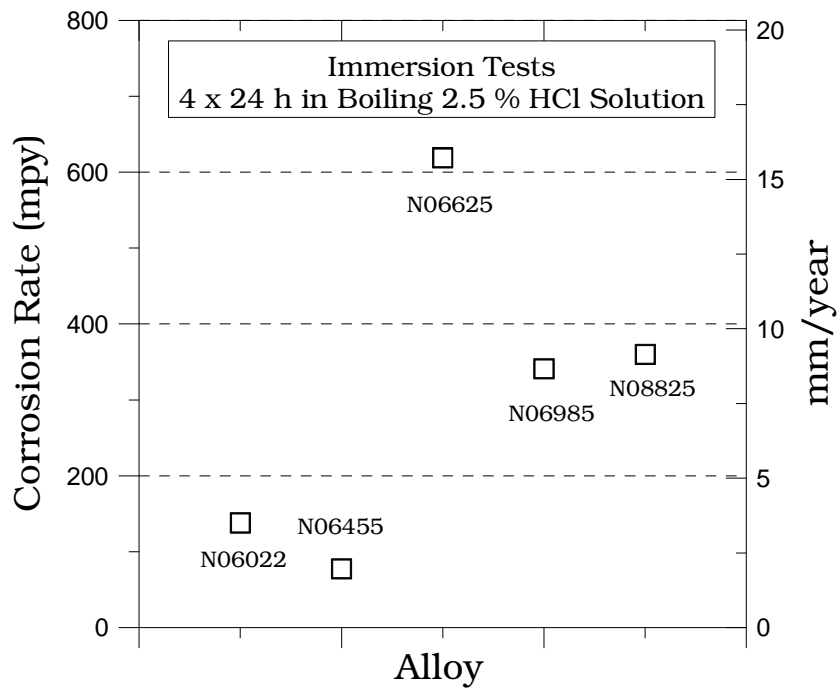


FIGURE7:CorrosionRateofthefivenickelalloysinboiling2.5%hydrochloricacidsolution.